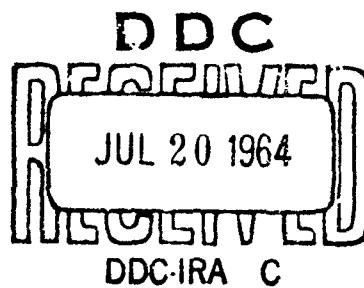


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TECHNICAL REPORT

WEIGHT LOSS OF PLATED METALS HEATED IN VACUUM

By

R. H. Wolff

Department of the Army Project No. 1-A-0-13001-A-039

AMC Code No. 5016.11.844

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Approved by:

A. C. Hanson
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Laboratory Director

24 January 1964

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

A series of experiments were conducted with 0.5 mil plated coatings on steel exposed to heat in vacuum. The plated coatings were cadmium, zinc, lead, nickel, and tin. Test conditions were exposure to vacuum at 1.5×10^{-5} torr and temperatures from 200° to 675°F . Periodic weight change measurements were made in room atmosphere using an analytical balance. Only slight variations of weight were observed with tests of lead, nickel and tin, and no significant losses under the conditions used. At their respective melting points, lead and tin softened and the coatings coalesced to form drops at the edges of the specimens.

Cadmium volatilized at rates of 3.0 to 7.6 mgm/cm²/day for temperatures at 400°F . By reference to initial coating weights, the cadmium coatings were completely evaporated.

Zinc coatings behaved in an unusual manner. An initial rate of weight loss occurred at 435° only to decrease to zero at 450°F with apparent constant weight. With increased temperature a rate of loss was resumed and rates of 4.5 to 6.5 mgm/cm²/day occurred at 550°F to a total loss of 90% coating weight. No further loss was observed through 675°F . By reference to initial coating weights, the zinc coatings were not completely evaporated. Plateaus of constant weight predominated at 450° although shallow slopes were noted at 475°F . Weight losses at these points varied from 22% to 30% of initial coating. Another phenomenon observed was the formation of a powdery layer when specimens were started at test temperature of 450°F . When brushed, this powder accounted for approximately 11% of coating weight and although substantial zinc was still present, further test and brushing gave no more powder loss. Time did not allow the identification of the powder and study of the surfaces.

RECOMMENDATIONS

Several avenues of exploration have been suggested by the results of this work. The effect of plating solution cyanide concentration on plated coatings in vacuum and the study of the surface changes of the zinc are particularly recommended for future work. On the basis of resistance to evaporation effects, it is recommended that zinc be used rather than cadmium as a protective coating for hardware, etc., that may be subjected to moderate temperatures in high vacuum.

WEIGHT LOSS OF PLATED METALS HEATED IN VACUUM

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WEIGHT LOSS OF PLATED METALS HEATED IN VACUUM

OBJECT

To determine the weight loss of plated coatings subjected to heating in vacuum.

INTRODUCTION

Interest in space exploration, as well as the increasing ability of vehicles to approach 100 mile altitudes has (1) required knowledge of behavior of materials in vacuum. Pressures at these altitudes are in the order of high vacuum (10^{-4} to 10^{-6} torr). (2) Except for occasional recorded determinations of vapor pressure in critical tables and handbooks, very little useable data has been available for the evaluation of metals and their behavior in vacuum. By application of thermodynamic principles, estimations of vapor pressures and evaporation effects for various metals and temperatures have been made. (3,4)

These calculations indicate that several metals, notably cadmium, zinc and magnesium, will sublime readily in the vacuum conditions of space. The actual effects will depend upon the temperature, pressure, and time. For extended periods of months or years in space these materials are inadequate for construction or protection as indicated theoretically. However, cadmium and zinc are two of the most effective protective metal coatings available for use on the ground. Recognizing that many items will be required to spend their major service life on the ground, perhaps alternating with projections into and recovery from high vacuum, interest was generated in the experimental behavior of these metals at moderate temperatures and short exposures in vacuum.

From a previous project (5) it was known that protective behavior of cadmium and zinc was not appreciably lessened by heating in vacuum except as temperatures were high enough to accelerate evaporation of the metals. Information was sought in this work in regard to the rate and completeness of metal loss.

EXPERIMENTAL PROCEDURE

Mild steel planchets 1/16 inch thick by 1 inch diameter were vapor degreased, electro-alkaline derusted, weighed, plated, weighed again, and then subjected to the vacuum test conditions. Plated coatings were cadmium, lead, nickel, tin, and zinc. Solution compositions for deposition of these metals are summarized in Table I. Specimens were prepared by

plating in triplicate to a thickness of approximately 0.5 mil, although the deposit weight was used as the test parameter.

Test conditions were imposed by placing the specimens in a vacuum chamber and heating the chamber to a selected temperature. The average vacuum condition was 1.5×10^{-5} torr. Exposure periods were regulated in two patterns. In the first pattern heat was applied for the full 24 hour period with only sufficient time out of test for weight determinations (less than an hour). The second pattern involved heating for approximately 8 hours with cooling in the vacuum for the remainder of the 24 hour period before weight measurement. Weighing was done in room temperature atmosphere using an analytical balance.

Tests were accomplished by starting at a selected initial temperature and raising the temperature periodically as changes were observed to occur (or not occur). The widest range used began at 200°F and ended at 675°F after a total of 53 days of exposure. Shorter duration tests were run at temperatures between 400° and 550°F.

EXPERIMENTAL RESULTS

Continued exposure of specimens at periodically increased temperatures resulted in no appreciable weight changes of the lead, tin, or nickel coatings. At their melting points the tin and lead coatings melted and coalesced at the bottom edges of the specimens. Other than slight darkening, which appeared to be a characteristic of the bright nickel plate at higher temperatures, the nickel coatings were unaffected throughout the 675°F temperature range.

The cadmium coatings showed an initial 24 hour weight loss* of 1% at 375°F, and continued to lose weight at a progressively increasing rate (see Figure 1). Increasing the temperature to 400° and then 435°F caused a further increase in rate. Maximum rates of 3.0 to 7.6 mgm/cm²/day occurred at 400°F. Some specimens lost 99% of the initial coating weight in approximately 6 days after appreciable weight loss began. The specimens plated from the low cyanide solution showed a slower rate of loss than those plated from the high cyanide solution. In both cases the specimens returned to initial uncoated weight, indicating a complete loss of coating.

* All weight losses are in terms of initial coating weight.

TABLE I
 COMPOSITION OF PLATING SOLUTIONS

SOLUTION	Make-Up or Analysis
	oz./gal g/l
Cadmium (Low)	Cadmium 3.74 28.0
	Cyanide 12.00 90.0
Cadmium (High)	Cadmium 2.71 20.3
	Cyanide 17.76 133.0
Zinc (Low)	Zinc 4.65 34.8
	Cyanide 10.97 82.2
Zinc (High)	Hydroxide 10.0 75.
	Zinc 4.85 36.4
Lead	Cyanide 14.23 106.7
	Hydroxide 10.0 75.
Lead	Lead 17.0 127.2
	Fluoboric acid 5.4 40.5
	Boric acid 3.0 22.5
Nickel	Nickel 9.3 69.6
	Chloride 2.4 18.0
	Boric acid 5.5 41.2
	pH-4
Tin	Sodium stannate 14. 105.
	Sodium hydroxide 1.25 9.4

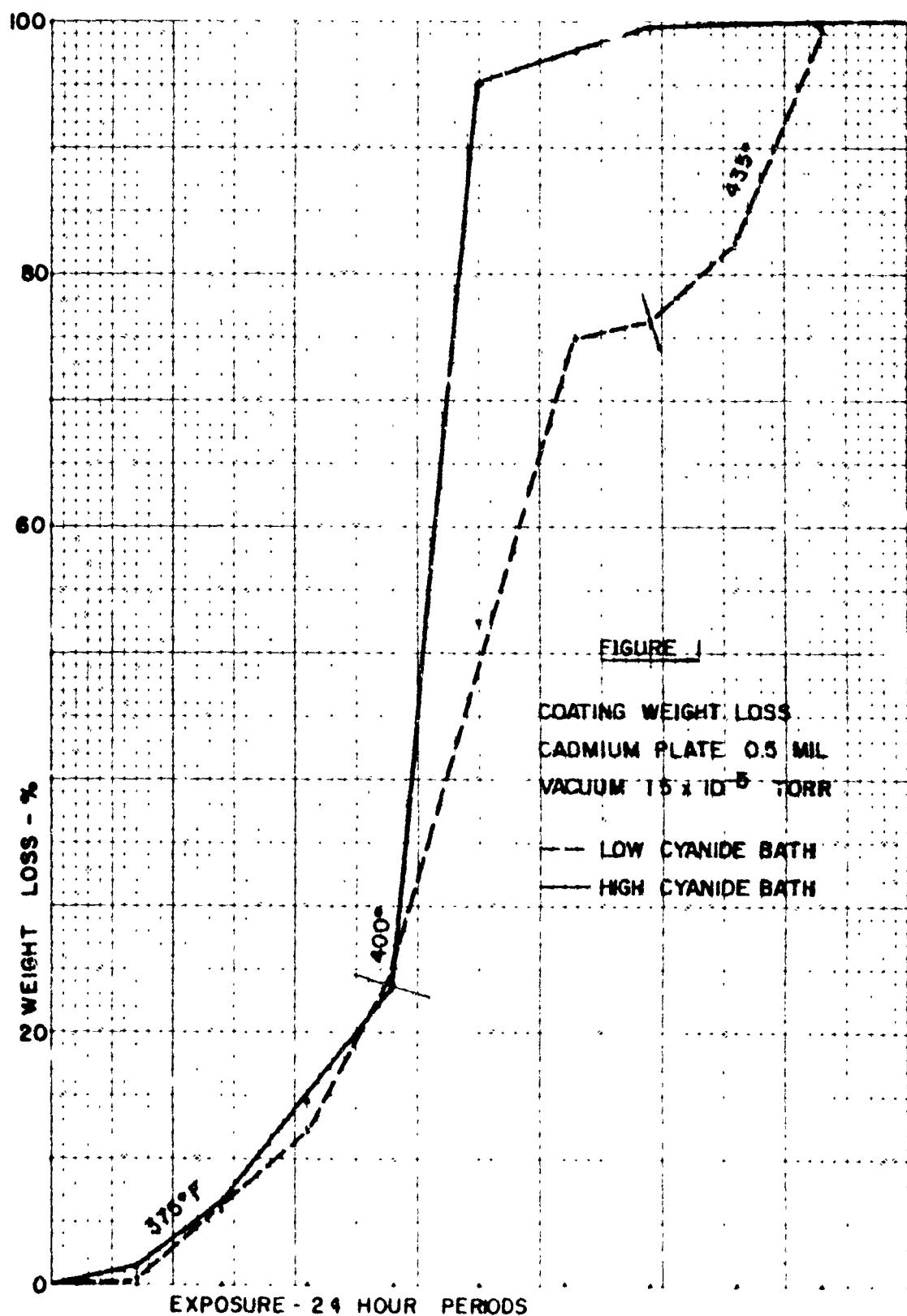


Figure 1

Zinc specimens behaved in a most peculiar manner. Coating weight loss of 1% in 24 hours occurred at 435°F . The rate of loss, however, decreased with continued exposure and dropped to almost zero. (See Figure 2). As the temperature was increased from 450° to 500°F , further weight losses were only minor. At 525°F the rate increased slightly. At 550°F a very rapid rate of 4.5 to 6.5 mgm/cm²/day resulted in 90% loss of coating weight. No further loss occurred for the duration of the test temperatures through 675°F .

The unusual behavior of zinc at temperatures between 400° and 550°F prompted tests to determine whether the change in rate was caused by interruption of the test for weight determinations. Zinc specimens were exposed to a continuous test of 24 hours in vacuum at 450°F . The average coating weight loss was $22 \frac{1}{2}\%$. No loss occurred at the second and third similar periods under the same conditions. A group of specimens heated under the same conditions for 6 hours lost 4.4% of weight. After 24 hours additional exposure the specimens had lost a total of 21.4%. This value remained the same after 16 hours of added exposure. See Table II and Figure 3.

During the next few tests some difficulty was encountered with spalling of portions of the high cyanide plate. Considerable time was lost in attempting to locate the source of the problem since no trouble had been observed in any of the previous tests. All solutions and procedures were checked. New solutions were finally prepared. Concurrently with the spalling difficulties it was observed that the non-spalled specimens appeared streaked. Brushing with soft brush removed a powdery smut. Specimens were then prepared, exposed at 450°F , weighed as tested, brushed, and weighed again. The initial weight loss by evaporation was approximately 19%; the secondary loss by brushing was 11.7% for a total loss of 30.7%. Upon continued heating at 450°F no further loss was observed.

In another test of 21 hours duration at the same temperature (450°F) specimens were brushed with similar results: 15.7% evaporation loss; 10.7% loss by brushing, for a total of 26.4%.

Specimens were exposed for 8 hour heat periods beginning at 400°F ; 450°F ; and 475°F . Weight determinations were made as removed from test and again after brushing the specimens. Results are tabulated in Tables III, IV, and V and are shown graphically in Figures 4, 5, and 6.

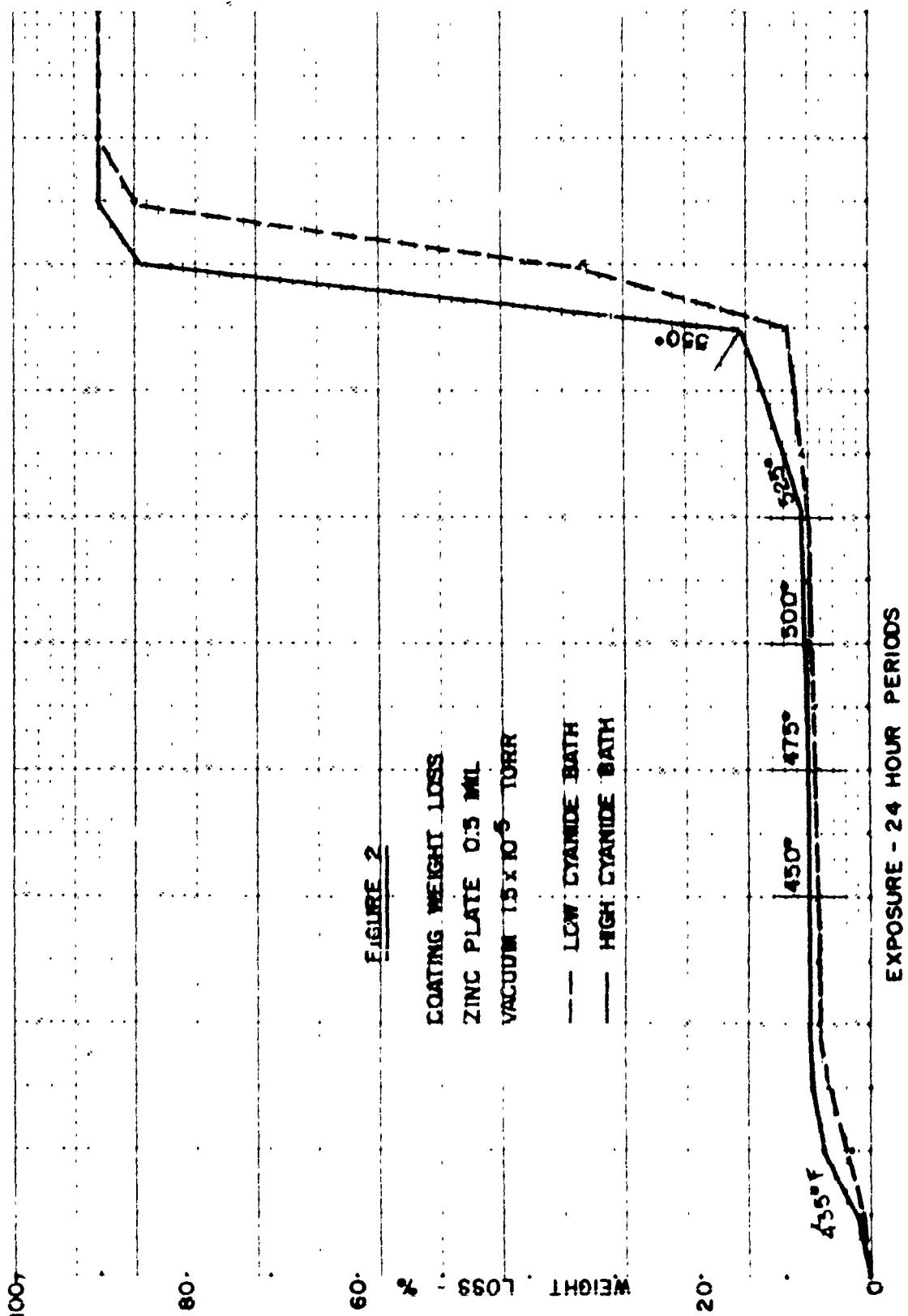


Figure 2

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TABLE II

Zinc Coating Weight Loss (.5 mil)

450° F 1.5×10^{-5} torr

Test	Heated Period (hours)	Weight Change (%)	Total Loss (%)
1	24	22.5	22.5
	24	0	22.5
	24	0	22.5
2	6	4.4	4.4
	24	17.	21.4
	16	0	21.4

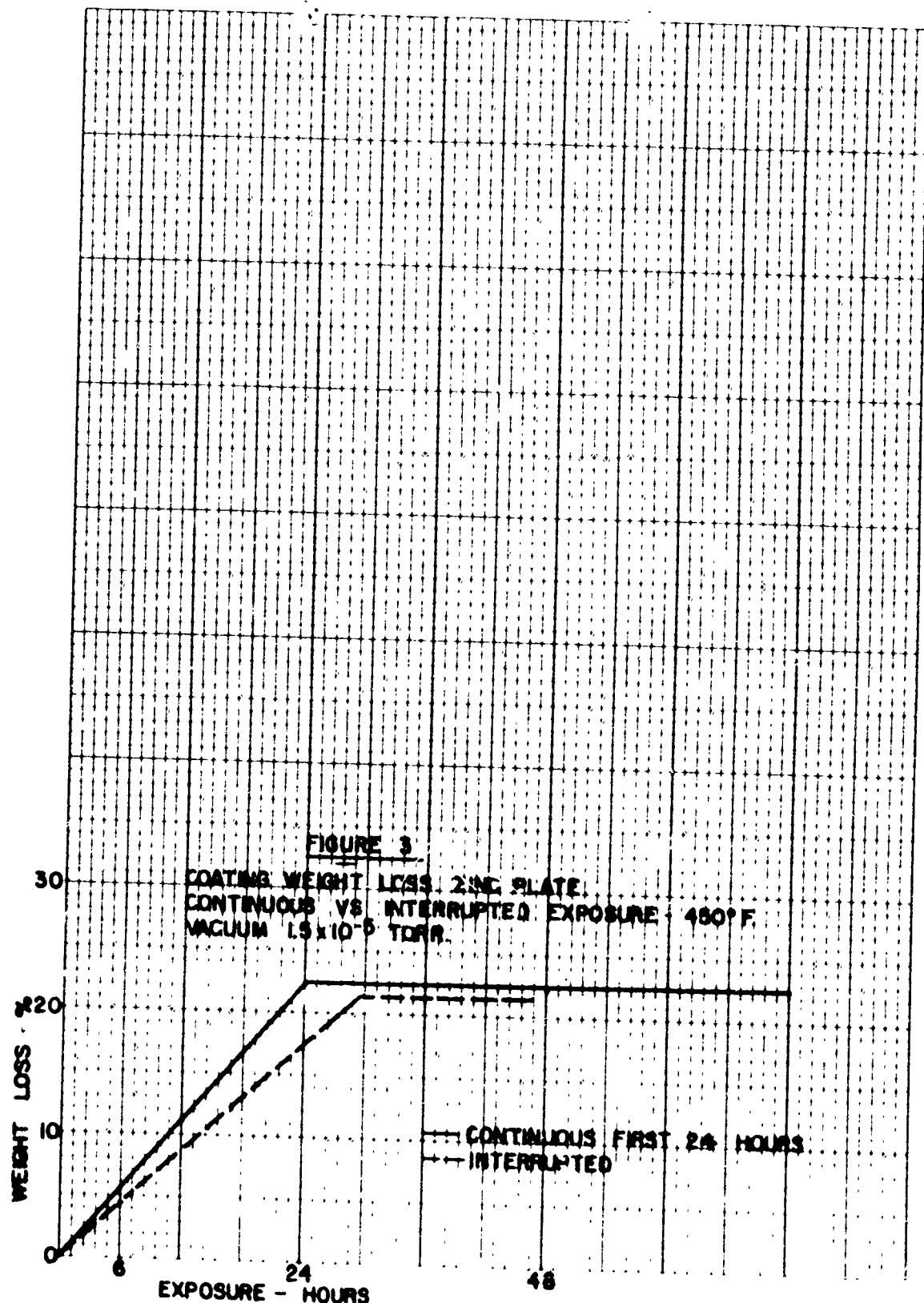


Figure 3

TABLE III
 WEIGHT LOSS OF ZINC PLATE
 INITIAL TEMPERATURE 400°F

TEMP. °F AVERAGE 8 HOUR PERIODS	% COATING WEIGHT LOSS		
	AS TESTED	AS BRUSHED	TOTAL
400	0	0	0
400	0	0	0
450	19.1	11.7	30.8
450	0	0	30.8
450	0	0	30.8
475	<1	0	
475	<1	0	31.7
500	0	0	31.7
500	0	0	31.7
525*	40.2	0	71.9
550*	17.8	0	89.7

TABLE IV
 WEIGHT LOSS OF ZINC PLATE
 INITIAL TEMPERATURE 450°F

TEMP. °F FOR 8 HR. PERIODS	% COATING WEIGHT LOSS		
	AS TESTED	AS BRUSHED	TOTAL
450	29.2	11.4	40.6
475	1.9	0	42.5
475	1.1	0	43.6
500	1.0	0	44.6
500	2.9	0	47.5
525*	48.5	0	96.
550*	0	0	96

* 23 hours

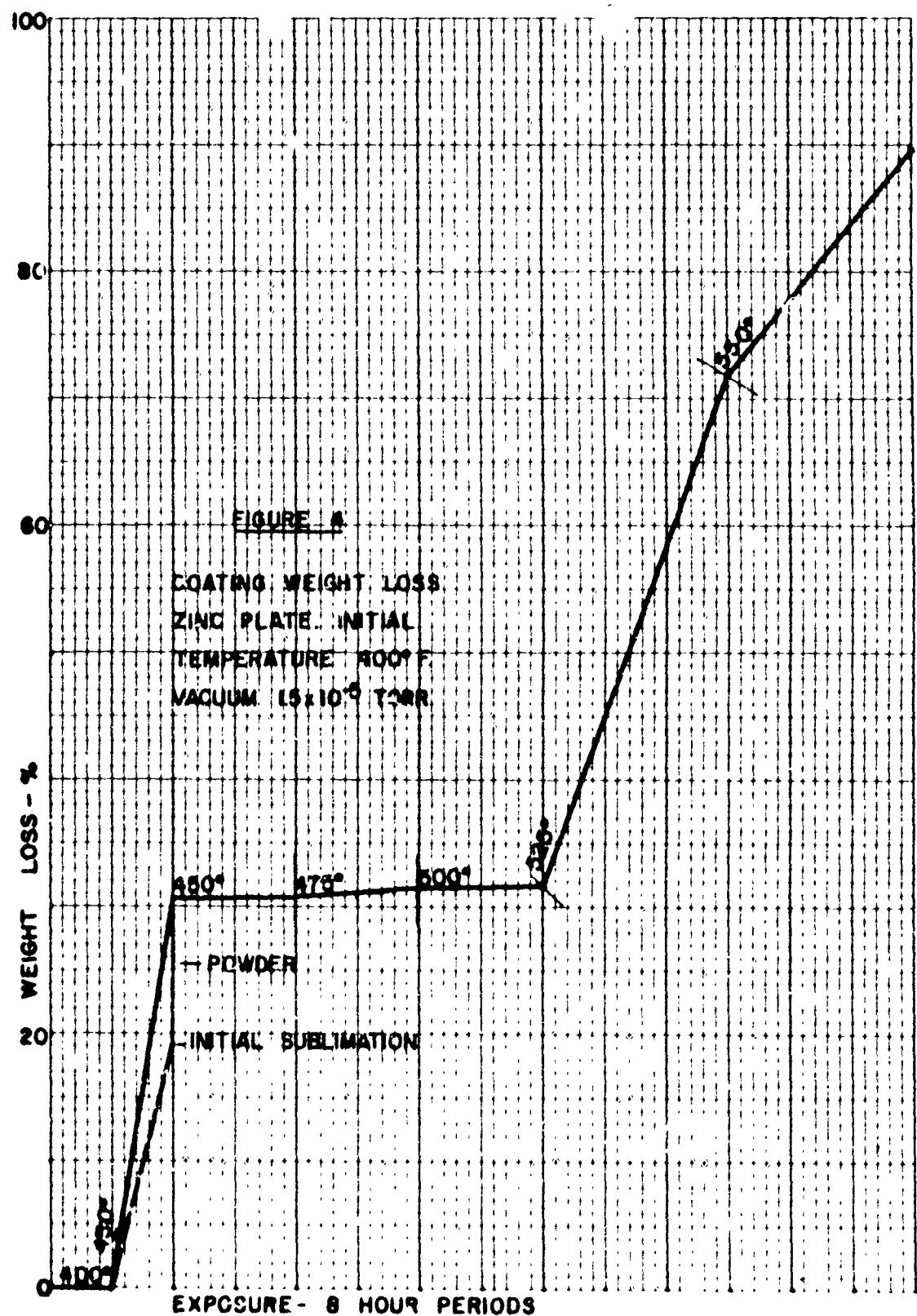


Figure 4

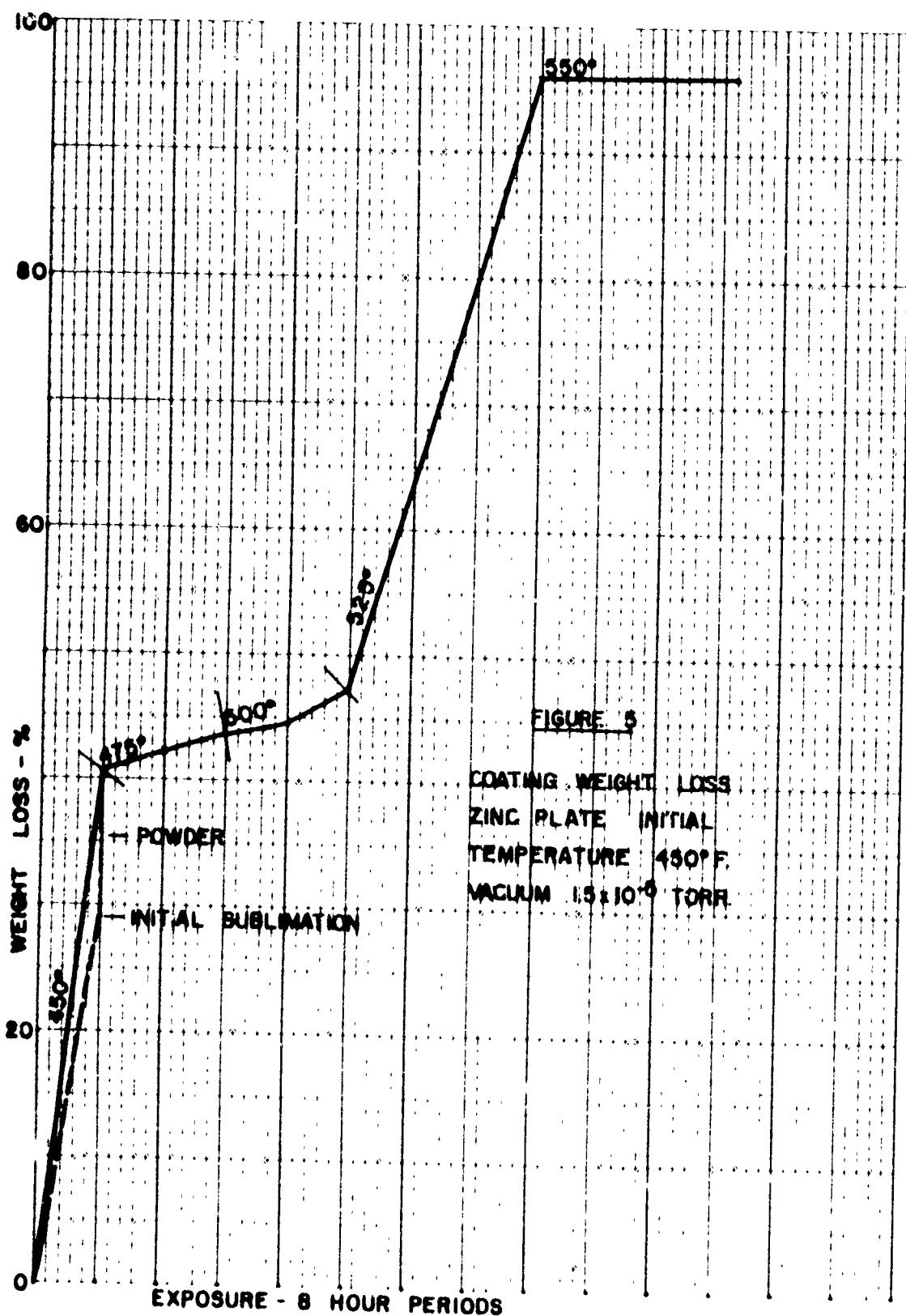


Figure 5

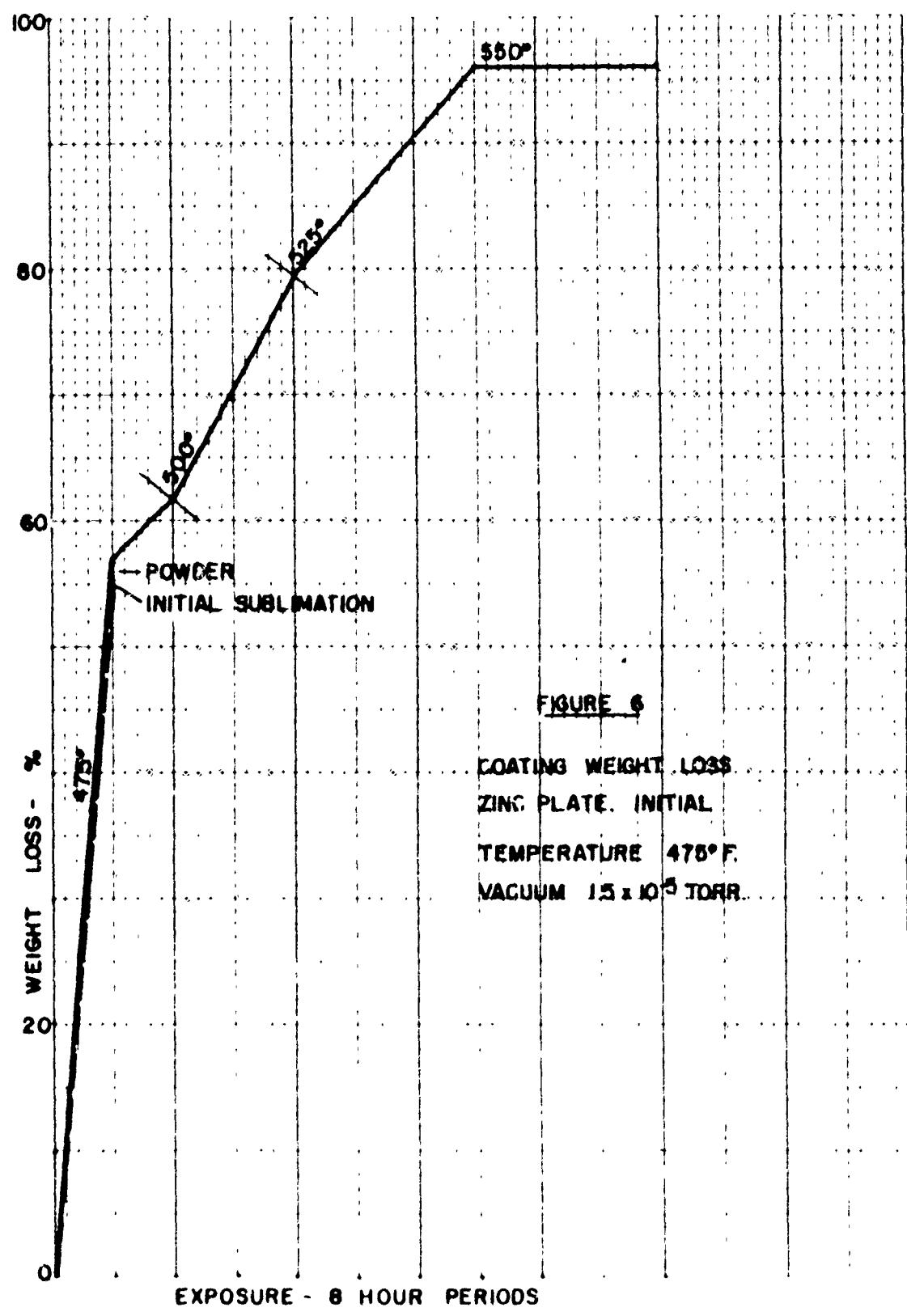


Figure 6

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TABLE V

WEIGHT LOSS OF ZINC PLATE
INITIAL TEMPERATURE 475°F

TEMP. °F FOR 8 HR. PERIODS	% COATING WEIGHT LOSS		
	AS TESTED	AS BRUSHED	TOTAL
475	55.	1.9	56.9
475	5.	0	61.9
500	7.8	0	69.7
500	9.9	0	79.6
525*	16.7	0	96.3
550*	0	0	96.3

* 23 hours

Appearance changes were slight for any of the metals up to 350°F. The cadmium plate developed pale yellow areas at 375°F which increased in size as exposure continued. At 400°F the cadmium specimens were almost completely yellow. This color remained until 475°F at which time a faint iridescent blue appeared.

The zinc specimens showed a slight difference in appearance between coatings from the low and high cyanide solutions in the test of longest duration. At 350°F the low cyanide specimens became dull and a fuzzy area developed at the bottom edge. This whisker appearance remained until the temperature reached 475°F. At 500°F, the general appearance became one of irregular black streaks which increased to a uniform cover at 550°F. The black color was presumed to be iron oxide resulting from periodic exposure of the specimens to the atmosphere for weighing.

Nickel coatings became tarnished at 550°F. This discoloration was believed to result from effects of organic addition agents used as brighteners in the plating solution.

The tin coatings melted and formed a small drop at the bottom of each specimen. The drop did not change for the remainder of the exposure through 675°F.

Lead specimens showed no appearance change until the coatings melted to form drops at the lower edges. No further changes occurred to 675°F.

DISCUSSION

Reviewing the course of the experiments in this work indicated that more questions were produced than could be dealt with systematically because of the time element. From theoretical considerations it was expected that graphical weight loss data would be accumulated which would result in approximately straight line curves of increasing slope for increasing temperatures. For cadmium, this statement was nearly true. For zinc the results were unexpected. With conditions of pressure and temperature below the minimum needed to produce a detectable weight change, the rate of change is, of course, zero. When the conditions are above this minimum it is expected that a rate will become constant as long as a volatile material is present and the conditions do not change.

When the apparent equilibrium condition appeared in the first test, more tests were undertaken at the approximate median temperature of 450°F. Assuming that early interruption of exposure to vacuum might result in a passive response, the tests were run comparing continuous and interrupted exposure. The parallel values and constant weight indicated that interruption was not a factor.

The weight loss observed at 450°F in a series of tests ranged from 20 to 30% on the basis of evaporation. When it was observed that a powder layer could be brushed away, the total loss became slightly higher. It is interesting to note that the powder did not recur after the initial removal, and appeared to be relatively constant (11%) for the coatings at 450°F. At higher temperatures the amount of powder was either less as formed or became volatile at these conditions. The powder does not prevent the evaporation effects, (See Figure 7) and therefore is not an explanation for the constant weight plateau.

It was also observed that the amount of loss (total) was about the same for different thicknesses of zinc at 450°F. Specimens plated at approximately 0.5 mil lost 29% of coating weight; specimens of 0.3 mil thickness lost 31% of coating weight. This suggests that the coating thickness is a factor either in the alloying effect with the specimen, or the conversion of the surface. If it is assumed that only a thin surface skin is converted to powder as other metal is evaporated, the percentage of weight loss should be much greater for a thin coating than for a heavier

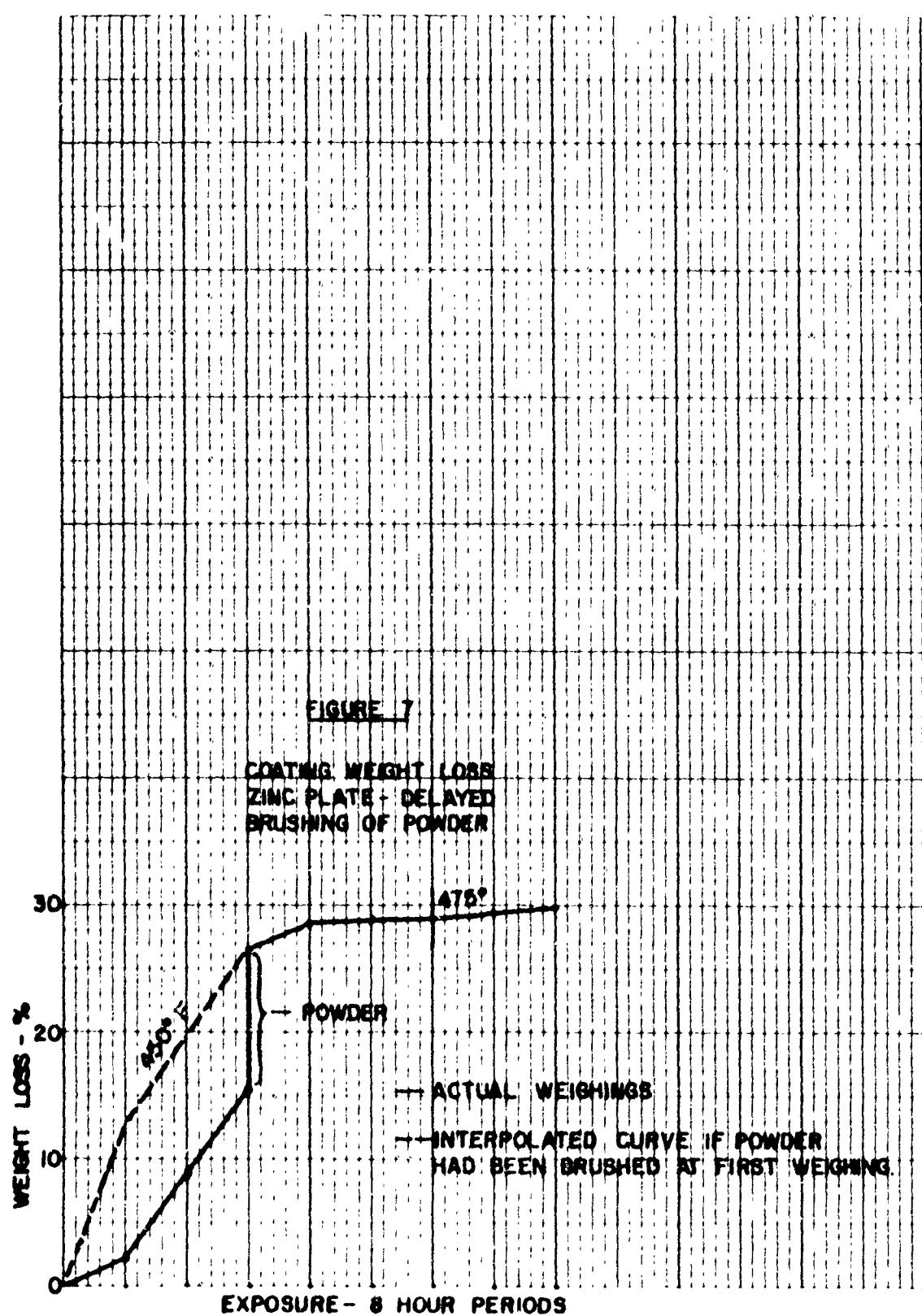


Figure 7

coating. If the assumption is made that for each unit of metal evaporated there is a residue of slag, so to speak, an explanation would be offered for the relation of different thicknesses of coating. This cannot be valid, however, because once completely brushed away the powder does not appear again. The powder is nevertheless related to the sublimation effects at 450°F since it does not appear at the 400°F tests which are below the detectable weight loss conditions.

The retention of 4 to 10% of initial zinc coating weight is not surprising, since zinc-iron-alloy layers are the basis of zinc cementation or "Sherardizing". Sherardizing is a diffusion in the solid state accomplished at temperature ranges of 660°F to 700°F and under certain circumstances may be done at temperatures above the melting point of zinc (787°F).⁽⁶⁾ Diffusion also occurs at temperatures lower than this and results in formation of iron zinc alloys which could account for the erratic and unusual evaporation effects observed. Formation of two layers of varying composition have been found.⁽⁷⁾ The extent of diffusion is dependent upon time and temperature, thus for higher initial temperatures, as suggested by the graphs in Figures 4, 5, and 6, the rate of evaporation probably increases more rapidly than the diffusion rate which forms the alloy. If this happens, the plateaus of lessened weight loss will occur at higher loss level. No completely satisfactory explanation is presently offered since analysis of the powder and study of the surfaces could not be accomplished at this time.

From the observations it appears that variations in behavior can be expected from coatings plated from solutions varying in cyanide concentration. For both cyanide soluble metals used in this work, cadmium and zinc, the rate of weight loss was slightly less for the plate from the lower concentrations of cyanide in the plating solutions. In view of reports of blistering or frothing of deposits in missile devices,⁽⁸⁾ this phase should be explored.

CONCLUSIONS

In consideration of usefulness of cadmium and zinc as extensions of protective coatings from earth atmospheres to high vacuum and moderate temperatures (350°F to 500°F) cadmium is of no practical value. The strange behavior of zinc suggests that for short period exposure it may offer practical use, at estimated temperature ranges less

than 450°F. The formation of an iron zinc alloy will offer protective ability even though a major portion of the zinc coating has been volatilized.

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illus., tables (DA Project No 1-A-0-13001-A-039
AMC Code No 5016 11-844) Unclassified report

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temperature a rate was resumed and rates of 4.5 to 6.5 $\text{mgs/cm}^2/\text{day}$ occurred at 550°F to a total loss of 90% coating weight. Zinc coatings were not completely evaporated. Weight losses at 450°F varied from 22% to 30% of initial coating. Another phenomenon observed was the formation of a powdery layer on specimens at 450°F. When brushed, this powder accounted for approximately 11% of coating weight and although substantial zinc was still present, further test and brushing gave no more powder loss. Time did not allow the identification of the powder and a study of the surfaces.

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